

AD 713222
AFCHL-70-0279

RESEARCH DIRECTED TOWARD THE
EXPERIMENTAL INVESTIGATION OF
METHODS OF ANALYZING $SO_2:SO_3$
RATIOS IN JET EXHAUSTS

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CONTRACT NO. AF19(620)-6137
PROJECT NO. 8379
TASK NO. 887901
WORK UNIT NO. 88790101

FINAL REPORT

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PREPARED FOR:

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OFFICE OF AEROSPACE RESEARCH
UNITED STATES AIR FORCE
BEDFORD, MASSACHUSETTS 01730

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ABSTRACT

This report provides a summary of the tasks, performance and equipment developed for Contract No. AF19(628)-6137.

Research was directed towards the analysis of SO_2/SO_3 ratios in jet exhausts at temperatures of 600°C and Mach I gas streams. Spectral absorbance measuring techniques in the ultra violet region of the spectrum appeared to offer the best method of measurement.

Equipment was designed and developed incorporating these techniques.

Other aspects of jet exhausts were also investigated including contrail scattering and ionization effects.

Equipment was also developed for the measurement of infrared radiation from a jet engine.

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Research was directed towards the analysis of SO_2/SO_3 ratios in jet exhausts at temperatures of 600°C and Mach I gas streams. Spectral absorbance measuring techniques in the ultra violet region of the spectrum appeared to offer the best method of measurement.

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I. CONTRACT OBJECTIVES

Tasks performed for this contract involve research directed towards the experimental investigation of methods of analyzing the ratio of sulfur dioxide to sulfur trioxide (SO_2/SO_3) in jet exhausts. The specific tasks performed were as follows:

- A. Study of means of determining SO_2/SO_3 ratios of temperatures of 600°C .
- B. Extension of the above high temperature studies to include SO_2/SO_3 ratios in Mach I gas streams containing air, carbon dioxide, and water in proportion to that found in jet exhausts with 60:1 air fuel ratios.
- C. Design, construct, test and supply a compact SO_2/SO_3 analyzer.
- D. Investigation of electromagnetic propagation of both back scatter and forward scatter of contrails, suppressed contrails and associated phenomena to include various SO_2/SO_3 ratios. This investigation included:
 - (1). The various parameters of dispersed particles, i. e., size, density, shape, structure with respect to the characteristics of radar systems.
 - (2). An examination of the phenomena of the scattering experienced by an electromagnetic wave from natural and artificially

produced contrails.

(3). Theoretical and practical aspects of aircraft exhaust composition, temperature, degree of ionization, particle size, shape, material, structure and density. The analysis was to include the Mie and the Rayleigh regions.

(4). Continuation of mathematical studies to further develop the mathematical model of electro-magnetic propagation in conjunction with natural and artificially produced contrails.

(5). Devise and assist in the execution and reduction of data for the experiments to verify or correlate the mathematical approach previously outlined.

(6). Theoretical analysis and calibration testing on an existing ionization chamber.

(7). Modify the ionization chamber apparatus to optimize its reading capability.

E. Redesign and modify a Coleman ESP-3T Recording Spectrophotometer to provide an Infrared Jet Exhaust Analyzer. The Spectrophotometer supplied by the Government was later changed to a Beckman DK-A Spectrophotometer.

II. PERFORMANCE OF TASKS

A & B. - Study of methods for determining SO_2/SO_3 ratios at temperatures of 600° and Mach 1 gas. -

Extensive research into measuring methods for SO_2 and SO_3 led the contractor to the conclusion that the best method for measuring these gases under the specified conditions was through the use of spectral absorption techniques.

Investigation of these techniques revealed that two spectral regions could possibly be considered: the infrared region of 7 - 8 μ and the ultra violet region of 230 - 250 $\text{m}\mu$.

Infrared

A literature search was conducted in order to obtain available information on absorption bands for SO_2 and SO_3 . Combining the spectral data for sulfur trioxide with the absorption spectrum of sulfur dioxide reveals a doublet absorption band that exists for both compounds, but that the trioxide is shifted to the lower wavelength. In theory, therefore, it should be possible to establish the ratio of the two compounds by measuring the absorption of the gas mixture at two or more selected wavelengths provided suitable resolution can be obtained and that the mixture is near a 1:1 ratio.

The infrared region was investigated experimentally using a Perkin-Elmer Model 21 Infrared Absorption Spectrophotometer, with a 10 cm cell length and NaCl windows. The absorption spectrum

(actually transmittance) of SO_2 at a pressure of 11 mm. Hg was plotted, showing the expected high absorption in a doublet near 7.3μ wavelength. The corresponding transmittance data was obtained on a Perkin-Elmer Model 257 Spectrophotometer. In both instruments, the spectrum was recorded on a pre-calibrated and pre-aligned chart whose drive is synchronized with the monochromator rotation. Upon attempting to measure the absorption spectrum of sulfur trioxide for comparison purposes, it was found that the same cells could not be used because of the chemical activity of the SO_3 on the sodium chloride windows. The compound is extremely reactive at low pressure. Accordingly, attempts were made to obtain an absorption spectrum of SO_3 employing germanium (Ge) windows in place of NaCl. These windows were selected because inertness was expected from the electronegativity location of the element in the periodic table. Experience proved, however, that the Ge windows also were etched by exposure to SO_3 . Windows of MgO later were found to be resistant to the deleterious effects of SO_3 .

Normalized curves, based on spectra obtained, show that SO_2 and SO_3 prime band spectral absorption characteristics should permit differentiation in the infrared region. According to calculations made based on perfect gas laws and pressure data available from literature (Refer to Quarterly Status Report No. 4, 15 May 1967 - 15 August 1967), it is theoretically possible to make the differentiation

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with a two thousand to one ratio in favor of SO_2 . However, it subsequently has been found that varying concentrations can result in one of the compounds blanking out the other. This is because the absorption bands in the infrared region are spaced so closely - approximately 30 wavenumbers (cm^{-1}) apart. In addition, it was found that interference could be encountered in the infrared region from other compounds such as water vapor and methane.

In view of the above, it was concluded that the use of absorption spectra in the infrared frequencies should be abandoned as a useful technique for determining the $\text{SO}_2:\text{SO}_3$ ratio in exhaust gases.

Ultraviolet

With the use of a Beckman DK-2A Spectrophotometer, the absorption spectra of SO_2 and SO_3 in the ultraviolet region 220 - 340 mu were recorded. A pronounced peak of absorption for SO_2 was found centered at about 285 mu.

A Perkin-Elmer Model 202 Spectrophotometer, a double beam instrument capable of operating in the ultraviolet - visible region was used to extend the range of investigation. The instrument was equipped with a 60-degree fused silica prism, tungsten filament and air-cooled deuterium arc source. It can be used in the 190 - 390 mu ultraviolet region. Quartz 151 windows were utilized for the cell. The spectrograms revealed that there is a marked absorbance for SO_2

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and SO_3 at wavelengths shorter than about 230-mu.

Subsequent tests of the Perkin-Elmer Model 202, the Jarrel Ash Co. spectrophotometer, and the Coleman Model 124 Hitachi double beam grating spectrophotometer led to a decision to incorporate the latter into a portable apparatus for determining $\text{SO}_2:\text{SO}_3$ ratios.

C. Design, construct, test, and supply a compact SO_2/SO_3 analyzer.

The contractor selected the Coleman Model 124 Spectrophotometer for the relatively small size of the unit, and its adaptability to the proposed operating system.

AFCRL personnel developed a method of coupling to the jet engine to provide the gas sample for analysis.

The contractor designed a mating gas input system that coupled directly to the output sampling system.

A one (1) micron filter was employed in the system to reduce the amount of solids that were emitted from the engine. A .7 micron filter was installed at a later date, but it was found to restrict the gas flow too much for satisfactory operation.

Thermocouples were provided at the input and output of the sampling cell for temperature sensing.

The sampling cell was designed and developed to fit within the Coleman unit in place of the existing sampling system.

A reference cell was also designed and developed to provide a gas substitution method of measurement, if the method was preferred by the operating personnel. However, this measurement technique is quite involved, and requires considerably more performance time. It may not be as effective under dynamic testing conditions as the methods described herein.

A Gilmont absolute and differential micrometric manometer was employed for pressure sensing across the system flow nozzle. The device features a single micrometer having 20 turns per inch allowing direct reading by adding the manometric displacement in both ends of the U-tube.

A blower and light baffle were developed for the evacuation of the gas from the system. The output was designed to feed the ventilating system at the AFCRL installation.

The instrument was checked out in the normal operating mode, and the system was assembled and delivered to AFCRL. Instructions were provided with the equipment.

Dynamic testing of the system could not be performed at the contractor's facility. However the contractor's personnel did participate in some of the initial testing at AFCRL.

Calibration of the instrument. - Calibration curves were run for each gas under controlled laboratory conditions. Measurements

were made at various different pressures within the spectral range of 185 - 400 mu. See Figures 1 and 2. In both cases a standardization against holmium glass was included. Calibration was based on the following considerations:

$$T = 10^{-abc} \quad (1)$$

where T = Transmittance

a = Extinction coefficient

b = thickness (cell length)

c = concentration (mols/liter)

and $A = \log \frac{1}{T}$

where A = Absorbance

hence $A = abc \quad (2)$

and $\frac{P}{PRT} = \frac{n}{V} = \frac{\text{no. of mols}}{\text{cc}} = c \quad (3)$

where P = Pressure in atmospheres

T = $^{\circ}\text{K}$

R = Gas constant

therefore $C = \frac{A}{ab} = \frac{P}{RT} \quad (4)$

or $ab = \frac{A}{P} RT \quad (5)$

At a fixed wavelength, the extinction coefficient should be constant, therefore

$$ab RT = \frac{A}{P} \text{ constant} \quad (6)$$

Absorbance values taken from the spectrograms must be corrected by deducting the absorbance of the quartz windows (the evacuated cell) from the total absorbance plotted at the particular pressure and wavelength. Care must be taken to measure from the zero absorbance baseline to the absorbance level for any specific pressure.

A study of the SO_2 curve reveals that SO_2 absorbs at two points within the 185 to 340 μ region. One absorbance peak occurs at approximately 285 μ . The other absorbance peak occurs at approximately 197 μ . The SO_2 curves are characterized by rather severe oscillations in absorbance with respect to wavelength. Because of the steep slope, these oscillations make calibration of absorbance versus pressure very difficult. Extreme care must be exercised in the selection of wavelength and absorbance values.

A study of the SO_3 curve reveals that SO_3 absorbs at the shorter wavelengths with peak absorbance occurring between 185 and 190 μ at the lower pressures. It should be noted that the oscillations are much lower in amplitude for a given concentration. These oscillations were found to be due to the conversion of SO_3 to SO_2 during the measuring process, and can not be completely eliminated because of the unstable nature of SO_3 . It should also be noted that SO_3 does not absorb in the region of 285 μ . The small amount shown on the graph is due to conversion of an extremely large concentration of SO_3 to SO_2 . However,

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at expected concentration levels, this would not occur.

Absorbance versus pressure can be plotted for any desired wavelength. The contractor selected 285 mu and 190 mu. 285 mu is near the peak absorbance point for SO_2 and 190 mu is near the peak for SO_3 . Three curves were developed; one at 285 mu for SO_2 ; one at 190 mu for SO_2 and one at 190 mu for SO_3 .

Measurements -

The absorbance reading taken at 285 mu for SO_2 can be converted to pressure by using the calibration curves. Once the pressure is known, the absorbance of SO_2 at 190 mu can be determined. This absorbance can be subtracted from the total absorbance measurement at 190 mu. The remaining absorbance is due to SO_3 . SO_3 pressure can be taken from the calibration chart and the SO_2 to SO_3 ratio determined.

Absorbance due to the cell windows must always be subtracted from the total absorbance that is measured, and zero absorbance must always be used as the base line.

Accuracy of calibration is restricted by the oscillatory shape of the absorbance curve with respect to wavelength and the small number of concentration or pressure levels available at any specific wavelength. Calibration could be made at a wavelength that is always at a peak absorbance value on the curve. Measurements could then be made precisely on the peak involved, instead of the slope of the curve.

Another method of calibrating for the SO_2 concentration would involve taking absorbance readings from the chart at a number of different wavelengths about the 285 μ wavelength, and developing a family of absorbance/pressure curves. Absorbance would then be measured at the different wavelengths; pressure values taken from the family of curves, and the average concentration determined. The average concentration would then be used to determine the absorbance of SO_2 at 190 μ , and subtracted from the total measured absorbance at that wavelength.

Practical operating considerations developed during testing at AFCRL include the following:

- (1). Gas flow should be shut off during the start up and shut down of the jet engine.
- (2). Runs should be limited to less than a five minute period to prevent the build up of deposits on the cell windows.
- (3). Absorbance of the empty cell should be measured before and after each run to ensure against the deposition of material on the windows.
- (4). If measured absorbance values are less than 1.0, the 0 - 1 Absorbance scale should be used to obtain the best scalar accuracy.
- (5). Slit width should be set to 0.5 μ .

(6). Instrument calibration and setup can be determined from the Instruction Book provided with the equipment.

D. Electromagnetic Propagation Investigation -

Initial effort in this area involved attempts to determine the type of particles most feasible for radio wave reflection. The three following type particles were studied:

- (1). Dielectric particles
- (2). Ionized crystals
- (3). Free electron and ionized molecules

Conclusions reached as a result of the initial investigation were as follows:

(1). The conductive ions should prove slightly better than the dielectric particles. To achieve substantial backscatter at micro-wavelengths, both types require very high particle densities. The amount of backscatter was found to be inversely proportional to the particle size.

(2). The achievement of practical amounts of electromagnetic energy appeared to be the most feasible in the presence of ionized molecules and free electrons.

Following the above initial conclusions, studies were conducted on the number densities of free electrons in ionized molecules necessary to produce signal reflections (reflection process similar to that found

in the ionosphere) from the particles. Ionospheric theory predicts the required particle density to produce complete reflection as:

$$N = \frac{4 \pi^2 m E}{e^2} f_o^2$$

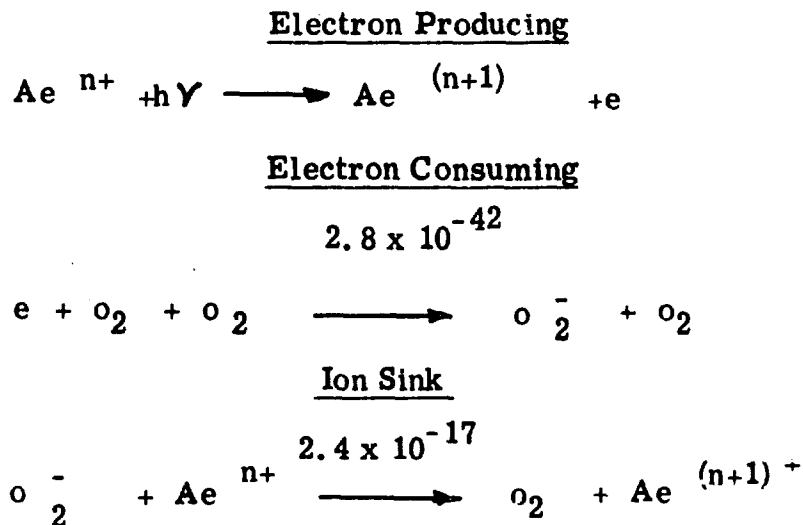
- where N = Particle density in particles/meter³.
 m = Mass of each individual particle in Kilograms.
 E = Permittivity of free space = 8.85×10^{-12} coulombs²/Newton meter².
 e = Electronic charge = 1.6×10^{-19} coulombs.
 f_o = Transmission frequency in Hz/second.

TABLE I

REQUIRED ELECTRON DENSITIES (N) FOR SELECTED
TRANSMISSION FREQUENCIES
 (Based on above formula calculations)

FREQUENCY (HERTZ)	ELECTRON DENSITIES (N _e) (Electrons/meter ³)
27 MHz	9.1×10^{12}
1 KMHz	1.25×10^{16}
10 KMHz	1.25×10^{18}

In addition to the preceding studies, available methods of producing plasmas with sufficient electron concentration to produce radio wave reflection were also investigated. In specific, this included studies performed to mathematically determine concentrations possible from UV irradiation of salt crystals. At atmospheric pressures, the usual chemical process for predicting the electron and ion concentrations is known as the "three-body-collision" process which is expressed as:



where Ae represents the NaCl particle.

For the "three-body-collision" process, the relation between electron production rate and the electron concentration is given by the expression:

$$\frac{dN_e}{dt} = q - \beta N_o^2 N_e$$

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where N_e = Electron concentration in electrons/meter³
 q = Production rate in electrons/meter³ seconds
 N_o = Oxygen concentration in molecules/meter³
 β = Electron attachment coefficient = 2.8×10^{-42}

At one-half atmosphere, $N_o = 2.68 \times 10^{24}$ molecules/meter³.

In a steady state condition, $dN_e/dt = 0$. Under these conditions, the expression relating production rate and free electron density becomes $q = 2.0 \times 10^7 N_e$ and the production rates for the frequencies previously noted in Table 1 are as listed in Table 2.

TABLE II
PRODUCTION RATES FOR SELECTED TRANSMISSION FREQUENCIES

FREQUENCY (Hertz)	ELECTRON DENSITY	PRODUCTION RATE (Necessary to sustain electron density)
27 MHz	9.1×10^{12}	1.82×10^{20}
1 KMHz	1.25×10^{16}	2.5×10^{23}
10 KMHz	1.25×10^{18}	2.5×10^{25}

Further studies concentrated on the development of mathematical predictions of relations between electron and ion densities in the AFCRL ionization chamber and electrical currents produced therein. Aspects considered in the chamber studies included:

(1) Resulting current from the chamber with an electron density of 5×10^9 electrons/meter³. Results showed that the following current values could be expected under the conditions noted:

Chamber electronic current, $I_e = 0.38 \times 10^{-10}$ amperes.

Chamber ionic current, $I_{O_2} = 9.24 \times 10^{-11}$ amperes.

Total current = 2.30×10^{-10} amperes.

where $N_e = 5 \times 10^9$ electrons/meter³.

$N_{O_2} = 3.2 \times 10^{12}$ ions/meter³.

V = Chamber applied voltage = 6 volts,

Flow rate of irradiated salt - air mixture = 0

and chamber dimensions of:

R_b , center electrode outer radius, = 0.50 inches

R_a , chamber inner radius, = 1.125 inches

L , chamber length = 10 inches

(2) Computations of the relative importance of free electron and ionized oxygen concentrations in air. For the conditions noted in the preceding paragraph (1), the following results were achieved:

Electronic current	=	1.28×10^{-10} amperes	=	0.60
Total current		2.30×10^{-10} amperes		

where: 60 percent of the current is due to electron movement and 40 percent to ion movement.

(3) Calculations of chamber current for a given production

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rate of 7.68×10^9 electrons/second and assumed complete attachment to O_2 molecules. With an aerosol mixture flow rate of 30 liters/minute, a chamber current of 5.22×10^{-12} amperes was obtained. With the flow rate cut-off, chamber current equals 1.2×10^{-9} amperes.

(4) Production rate necessary to produce a current of 10^{-9} amperes in a nitrogen atmosphere with a flow rate of 30 liters/minute. Results indicated $Q = 7.68 \times 10^9$ electrons/second.

Small Particle Scattering/Reflection Tests. -

Limited small scale tests were also carried out on the possibility of establishing a communications link between two sites using the technique of scattering from small particles. Previous theoretical studies had indicated the feasibility of this approach. The tests were performed at the rooftop radiation testing area of Barkley and Dexter Laboratories, Inc.

The following test equipment was assembled:

SHF signal generator, Hewlett-Packard model 620A,
output: 1 milliwatt (0 dbm)

Microwave receiver, Polarad model R,
sensitivity: 80 dbm

Microwave horn, 7.3 cm.

Microwave horn, 3.8 cm.

44" parabolic reflector and dipole feed

Flat conducting plate

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Flat board

Cloth screen

Aerosol powder (Al_2O_3)

Powder sprayer

Water

Salt water

The Fresnel range of the larger horn was calculated from the relation:

$$R = \frac{D^2}{4 \lambda}$$

where: R = Fresnel range in cm.

D = Aperture in cm.

λ = wavelength in cm.

and

$$R = \frac{(7.3)^2}{4 \times 3} = 4.44 \text{ cm.}$$

The above would indicate close placement of the equipment. A minimum range of 10 cm. was therefore assumed. On the basis of a 10 cm. range, the required radar cross section was calculated from:

$$\delta = (4\pi)^3 (P_r/P_o) (R^4 / \lambda^4 G^2)$$

where:

δ = radar cross section in square wavelengths

P_r = receiver sensitivity in milliwatts

P_o = transmitter power in milliwatts

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R = range in cm.

λ = wavelength in cm.

G = antenna gain

and

$$\begin{aligned}\delta &= (4\pi)^3 (10^{-8}/1) \left[10^4/3^4 (15)^2 \right] = 10.7 \times 10^{-6} \text{ sq. } \lambda \\ &= 9.6 \times 10^{-5} \text{ cm.}^2\end{aligned}$$

or

δ = a square 0.01 cm. on a side

The amount of powder in suspension was calculated on the basis of a 0.02 cm. radius conducting particle. The radar cross section of a single particle was calculated from:

$$\frac{d\delta(\theta)}{d\Omega} = a^2 (Ka)^4 \left[5/8(1+\cos^2 \theta) + \cos \theta \right]$$

where

$\frac{d\delta(\theta)}{d\Omega}$ = scattering cross section of conducting sphere

a = particle radius = 0.02 cm.

K = $2\pi/\lambda = 2\pi/3$

θ = angle between transmitter and receiver (assumed = 0)

and

$$\frac{d\delta(\theta)}{d\Omega} = 2.7 \times 10^{-9} \text{ cm.}^2$$

The number of particles (N) required determined as follows:

$$N = \frac{\delta \text{ required}}{\delta \text{ per particle}}$$

$$= \frac{9.6 \times 10^{-5}}{2.7 \times 10^{-9}}$$

$$= 3.6 \times 10^4 \text{ particles}$$

For this size particle there are 5×10^6 particles per pound.

Therefore, the pounds of particles per cloud calculated to be:

$$\text{pounds in cloud} = \frac{\text{particles/cloud}}{\text{particles/pound}}$$

$$= \frac{3.6 \times 10^{-4}}{5 \times 10^6}$$

$$= 7.1 \times 10^{-3} \text{ pounds.}$$

The experiment results are summarized in Table 3.

TABLE III

Experiment No.	Description	Results
1	Horns side by side - aerosol	Negative
2	Horns facing each other - aerosol	Negative
3	Bouncing off flat plate - aerosol	Negative
4	Horn parabolic reflector - aerosol	Negative
5	Horns side by side - water spray	Negative
6	Reflection of metal plate	23 db.
7	Reflection of flat board	6 db.
8	Reflection of cloth screen	0 db.
9	Reflection of cloth screen with aerosol powder sprinkled on	6 db.

TABLE III (Con't.)

Experiment No.	Description	Results
10	Reflection of cloth screen with water	0 db.
11	Reflection of cloth screen with salt water sprayed on	23 db.
12	Conductivity measurement of particles	zero conductivity

In addition to the aforementioned work, the contractor provided personnel to perform theoretical analysis and practical modification for the ionization chamber apparatus as requested by AFCRL personnel.

E. Redesign and modify an infrared spectrophotometer for analysis of jet exhausts -

A government furnished Beckman DK-A Infrared Spectrophotometer was modified by the contractor to permit the examination of the infrared spectrum radiated by a jet engine.

Radiated power levels could not be predicted. The contractor mocked up a lead sulphide cell in a holder mounted within a cylindrical tube to make some basic power level measurements. Tests were then conducted at the test site, using the basic instrument.

Levels obtained were recreated through the substitution of another source at the contractor's facility. The source radiation was sampled with the Beckman DK-A Infrared Spectrophotometer to determine the range of operation.

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Operating distances were then considered, and the gain required was calculated. Based on the calculations, an optical system was designed to collect the necessary energy.

The system consists of a parabolic mirror and a primary reflector that focuses the collected energy on the input detection system of the spectrophotometer. The collected energy is simply substituted for the energy source located in the spectrophotometer.

A cylindrical shield and a gas feed system were constructed to mount in front of the parabolic mirror in an attempt to keep foreign matter from entering the optical system. Dry nitrogen or air can be fed into the system to keep the optics free of contamination. Dry nitrogen may also be required to purge the spectrophotometer.

The contractor has delivered this equipment to AFCRL.

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III. CONCLUSIONS

The tasks performed over the period of three and one half (3 1/2) years have been documented in Quarterly Status Reports 1 through 10. Details could not be completely covered in this summary report. It is suggested that requests for information be placed with the Project Monitor, Mr. Seymour J. Birstein, CRF.

NOT REPRODUCIBLE

EVACUATED CELL
HOLMIUM GLASS STD
WAVELENGTH 0-2.1 μm

400

502

360.9

HOLMIUM GLASS

750
1250

1.984

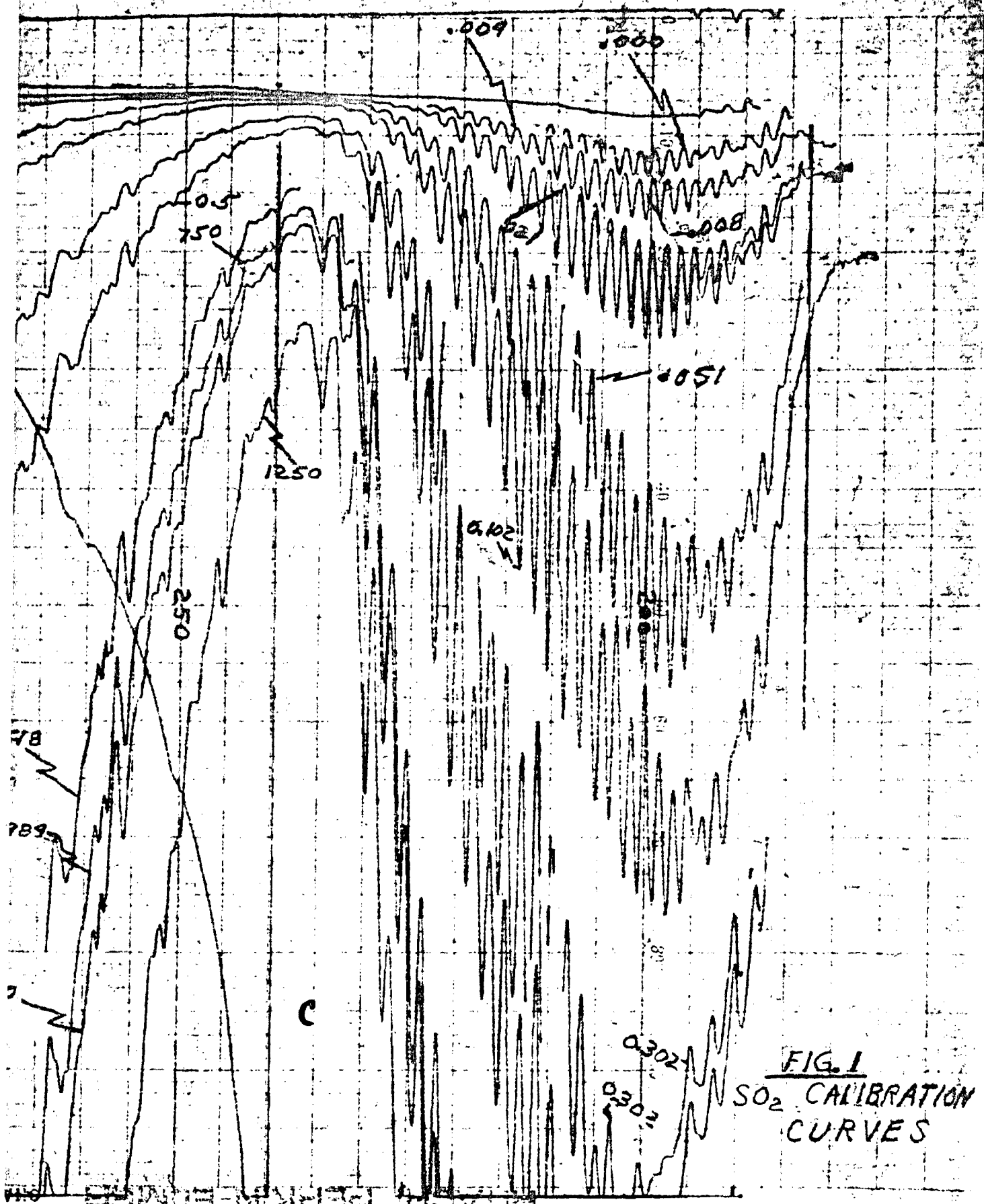
1.518
1.984

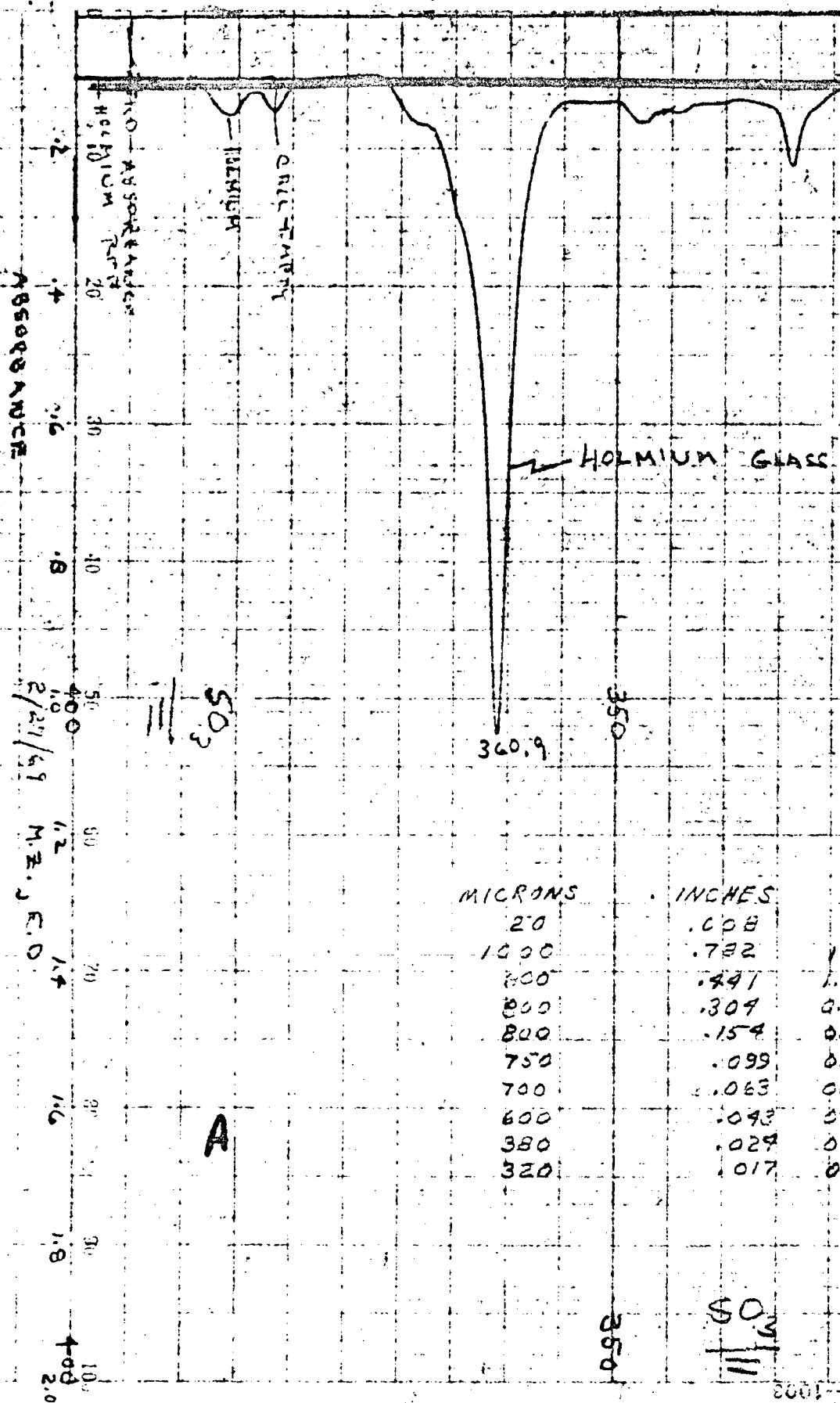
M.E.
E.D.

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MANOMETER (inches of Hg)	THERMOCOUPLE GAUGE (microns)
1.984	1000
1.518	750
0.500	750
0.302	750
0.102	635
0.051	555
0.021	350
0.008	340
0.004	218
0.000	159
-0.0025	100
-0.0035	60
-0.006	30

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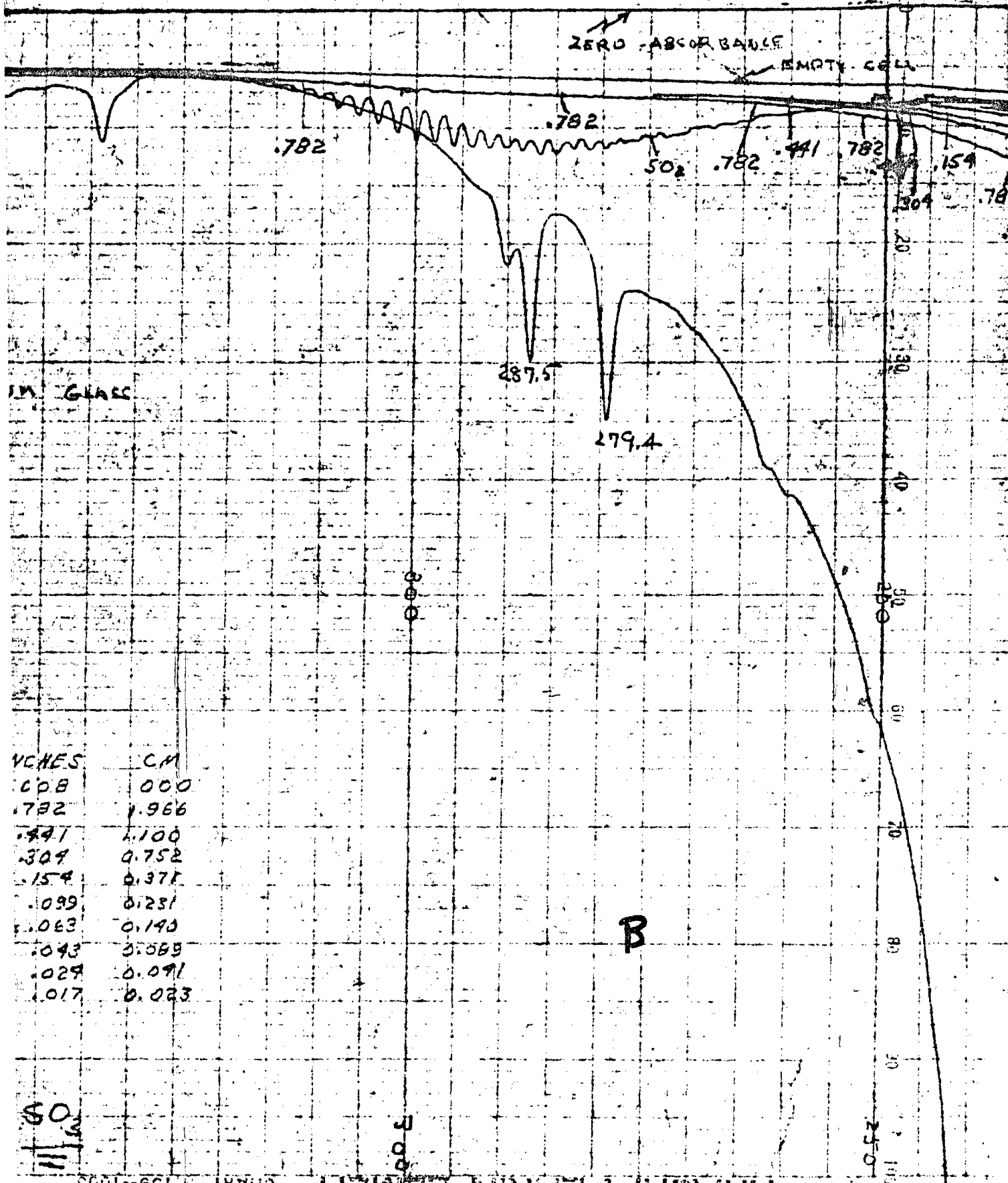




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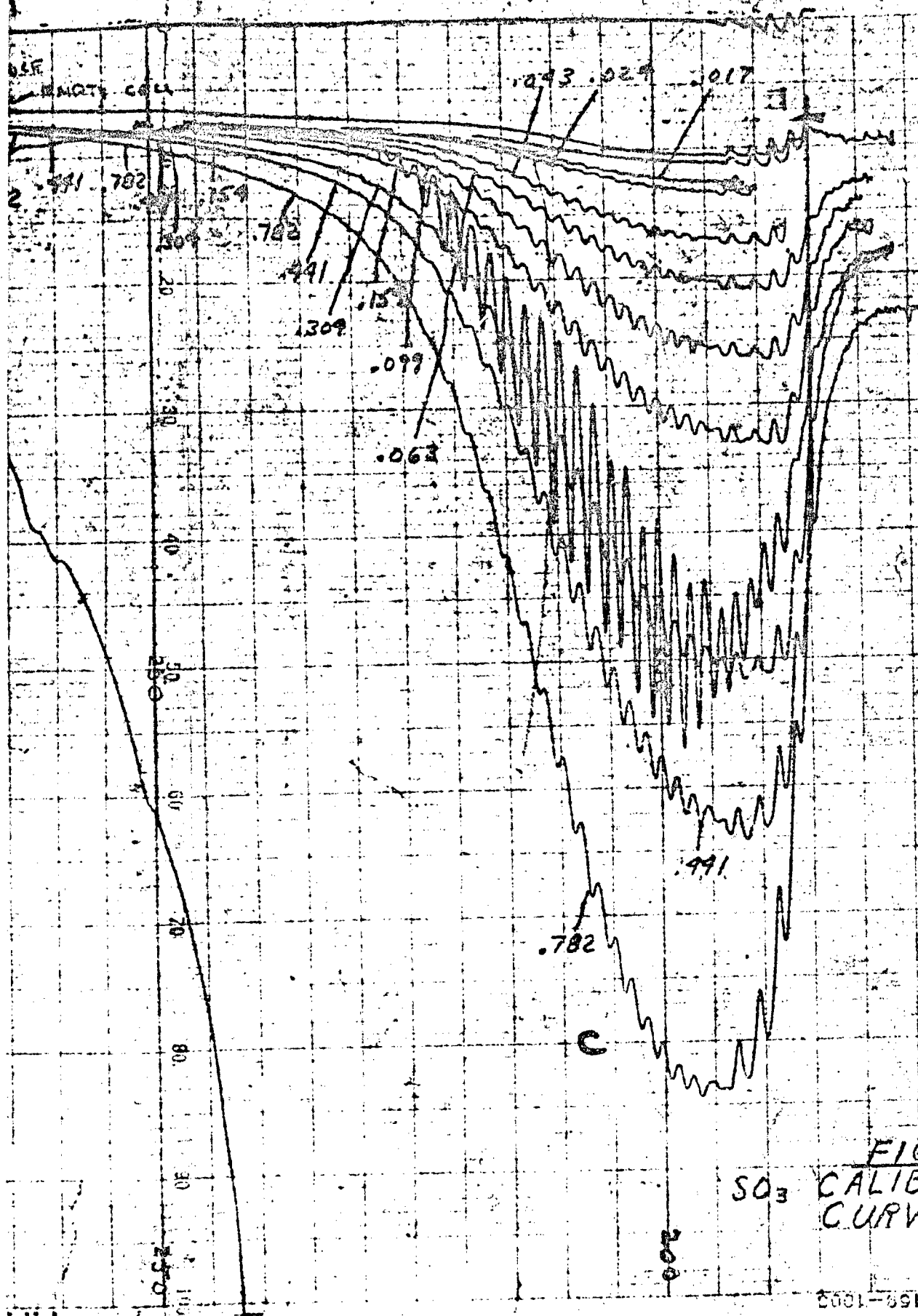
ZERO - ABSORBANCE

EMPTY CELL



CM-1	CM
4000	0.000
3000	0.000
2800	0.000
2000	0.000
1800	0.000
1600	0.000
1500	0.000
1400	0.000
1300	0.000
1200	0.000
1100	0.000
1000	0.000
900	0.000
800	0.000
700	0.000
600	0.000
500	0.000
400	0.000
300	0.000
200	0.000
100	0.000

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	ROLE	WT	ROLE	WT	ROLE	WT
Analysis						
SO ₂ /SO ₃ ratios						
Jet exhausts						
Infrared						
Ultra violet						
Sulfur dioxide						
Sulfur Trioxide						
Absorbance						
Electromagnetic propagation						
Spec.rophotometer						

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